

Figure 1. The UV spectrum of tetrodotoxin injection (990120-1A) identification

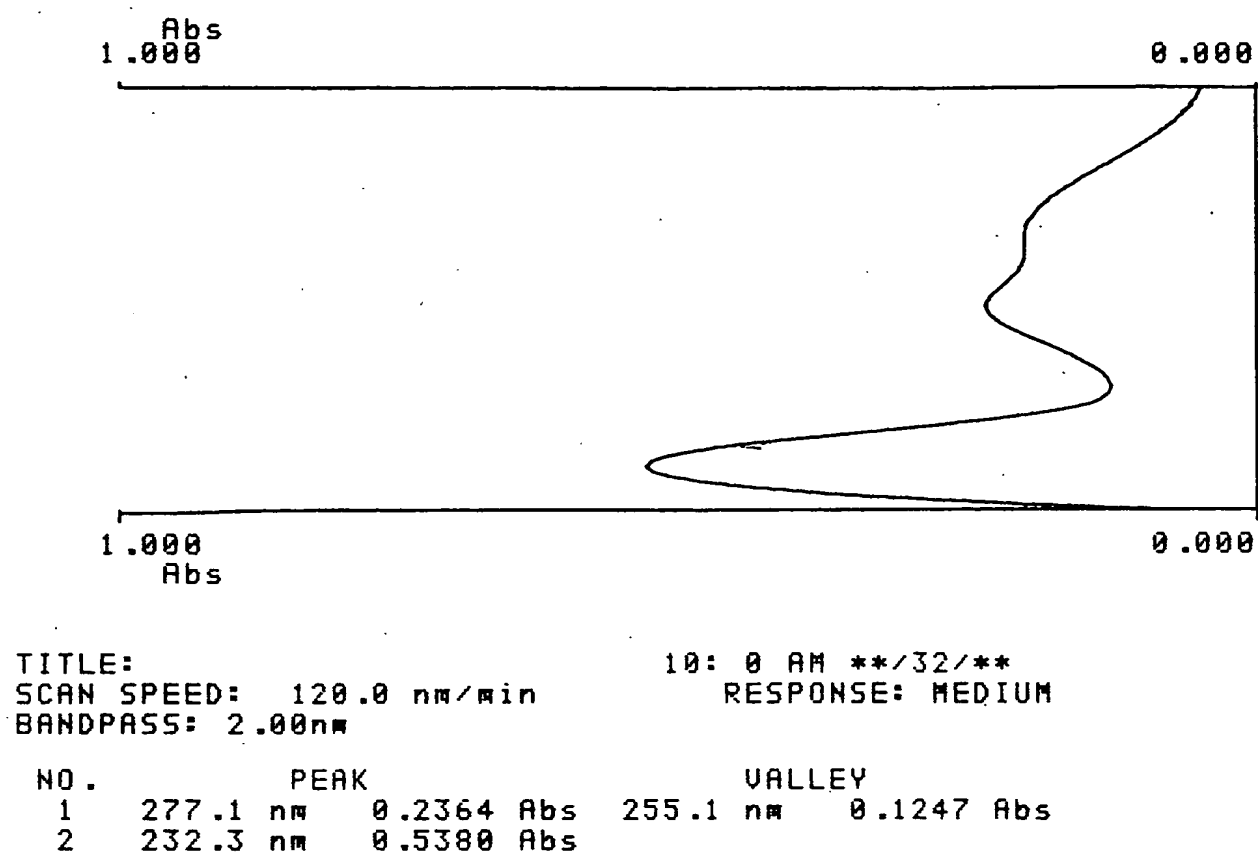
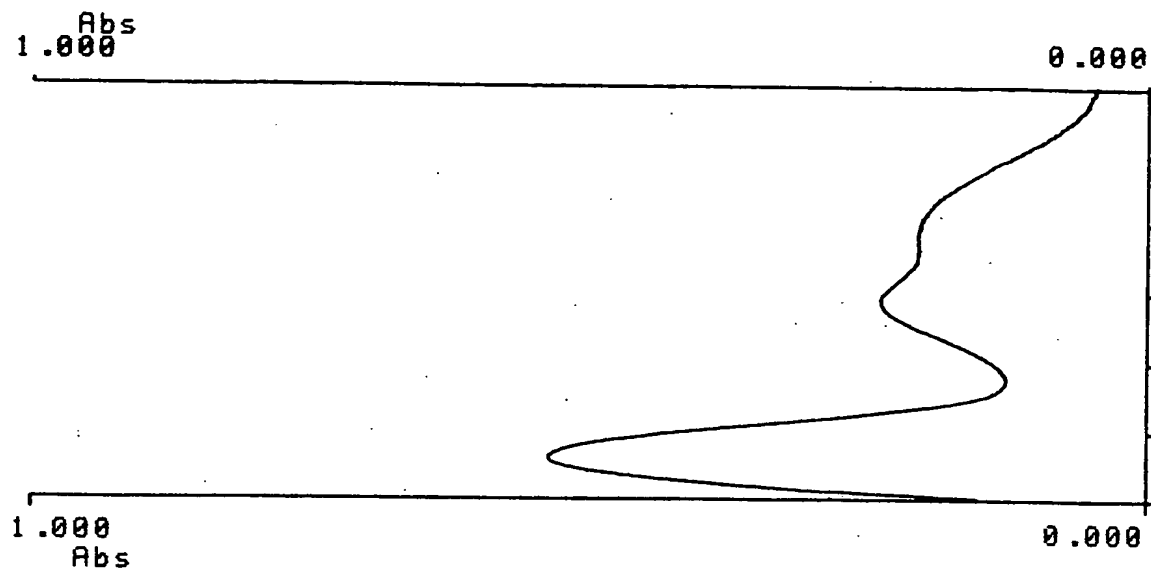


Figure 2. The UV spectrum of Tetrodotoxin injection (990121-2A) identification



TITLE:

SCAN SPEED: 120.0 nm/min

BANDPASS: 2.00nm

10: 0 AM \*\*/32/\*\*

RESPONSE: MEDIUM

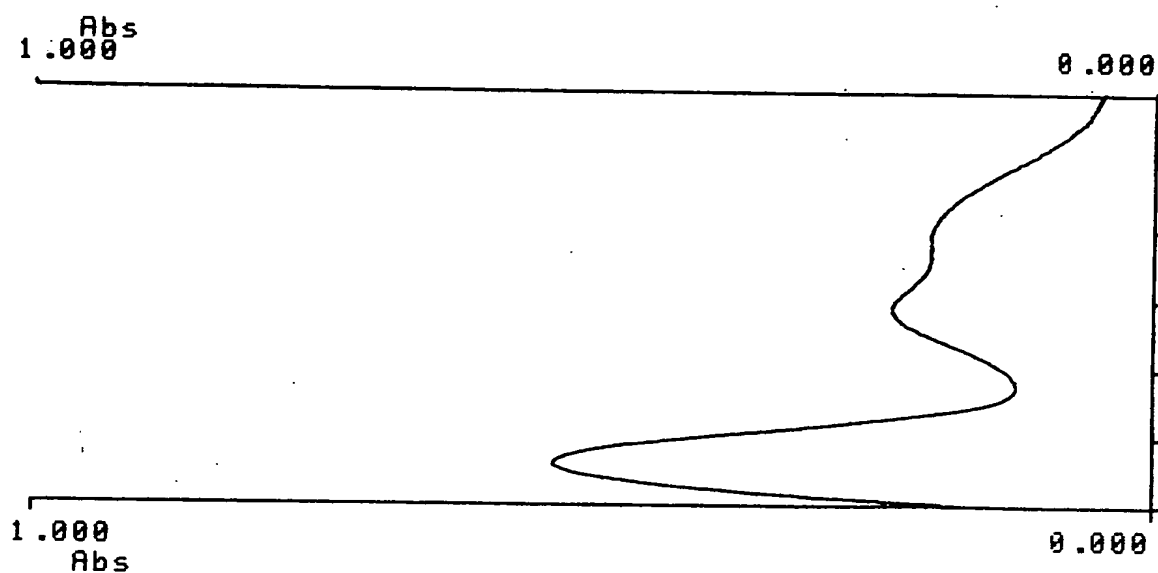
NO.

PEAK

VALLEY

1	277.6 nm	0.2357 Abs	255.6 nm	0.1243 Abs
2	232.8 nm	0.5365 Abs		

Figure 3. The UV spectrum of Tetrodotoxin injection (990122-3A) identification.



TITLE:

SCAN SPEED: 120.0 nm/min

BANDPASS: 2.00nm

10: 0 AM \*\*/32/\*\*

RESPONSE: MEDIUM

NO.

PEAK

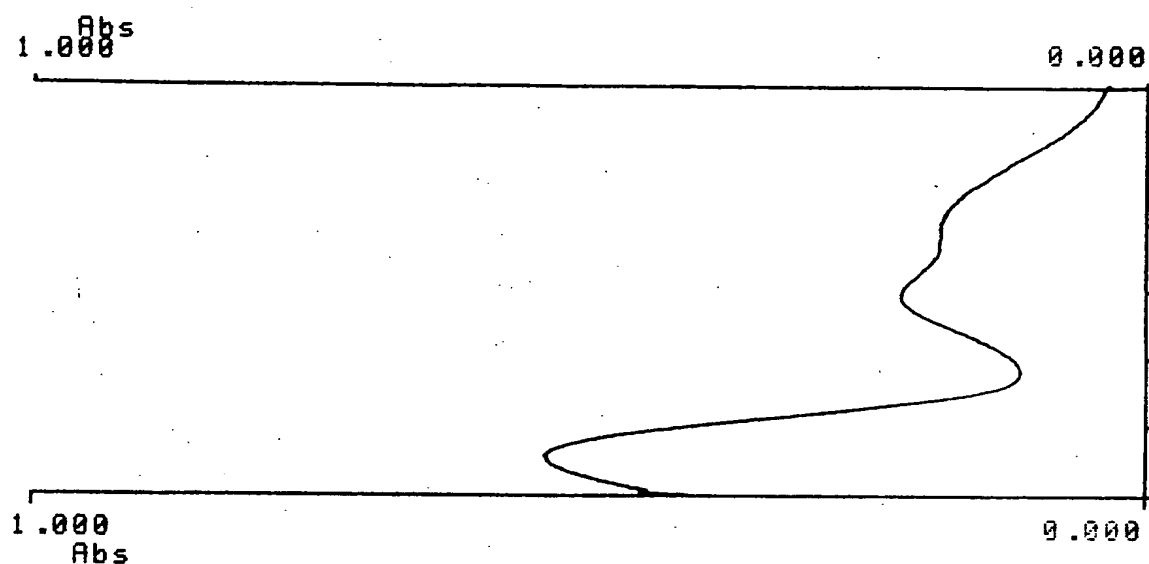
VALLEY

1 277.0 nm 0.2355 Abs

2 232.0 nm 0.5364 Abs

254.2 nm 0.1240 Abs

Figure 4. The UV spectrum of the Tetrodotoxin control sample (S-1) identification.

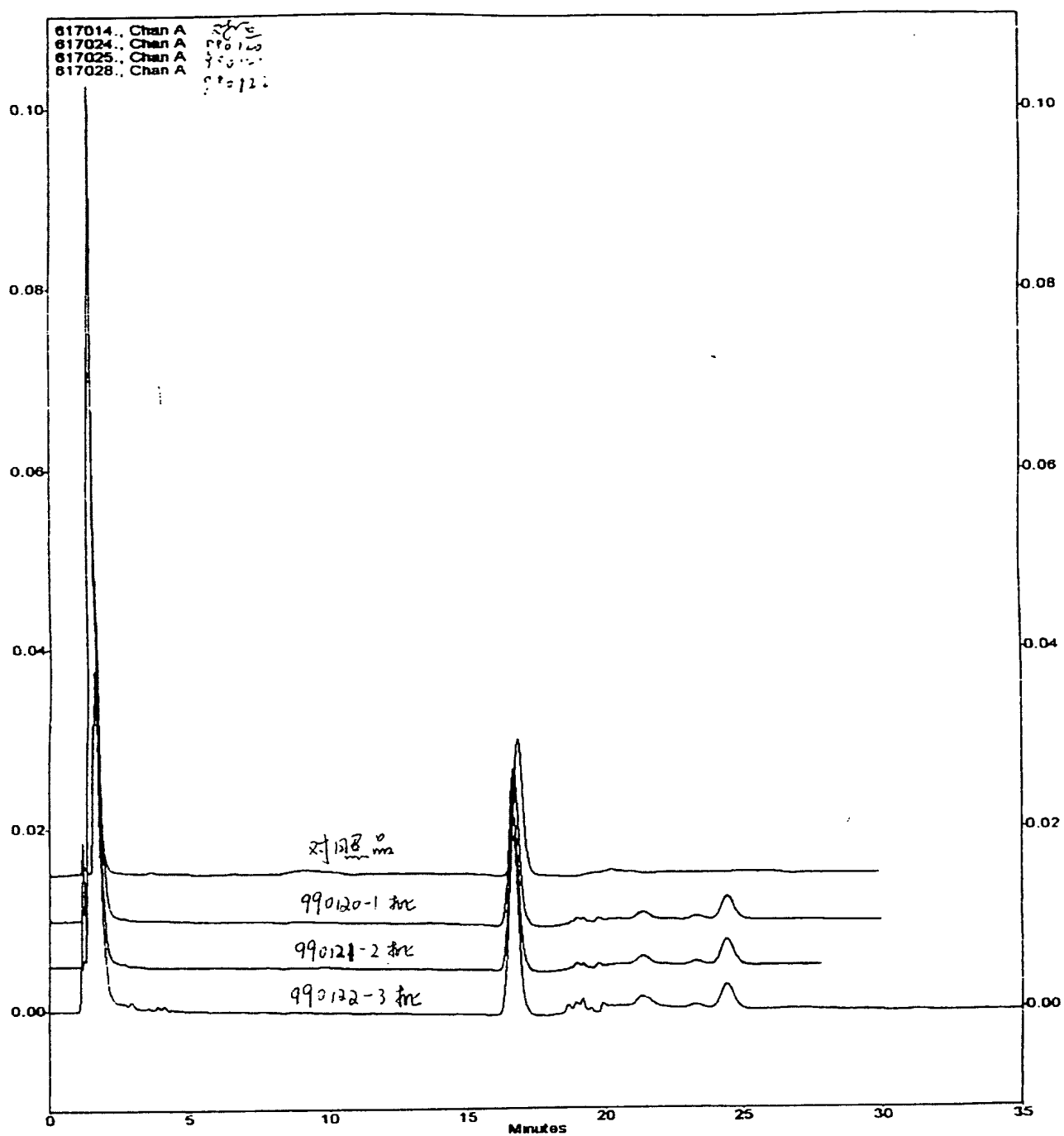


TITLE:  
 SCAN SPEED: 120.0 nm/min  
 BANDPASS: 2.00nm

10: 0 AM \*\*/32/\*\*  
 RESPONSE: MEDIUM

NO.	PEAK	VALLEY
1	277.5 nm 0.2174 Abs	255.7 nm 0.1120 Abs
2	230.8 nm 0.5393 Abs	

**Figure 5.** The chromatogram of the retention time of the three batches of tetrodotoxin and its control sample.



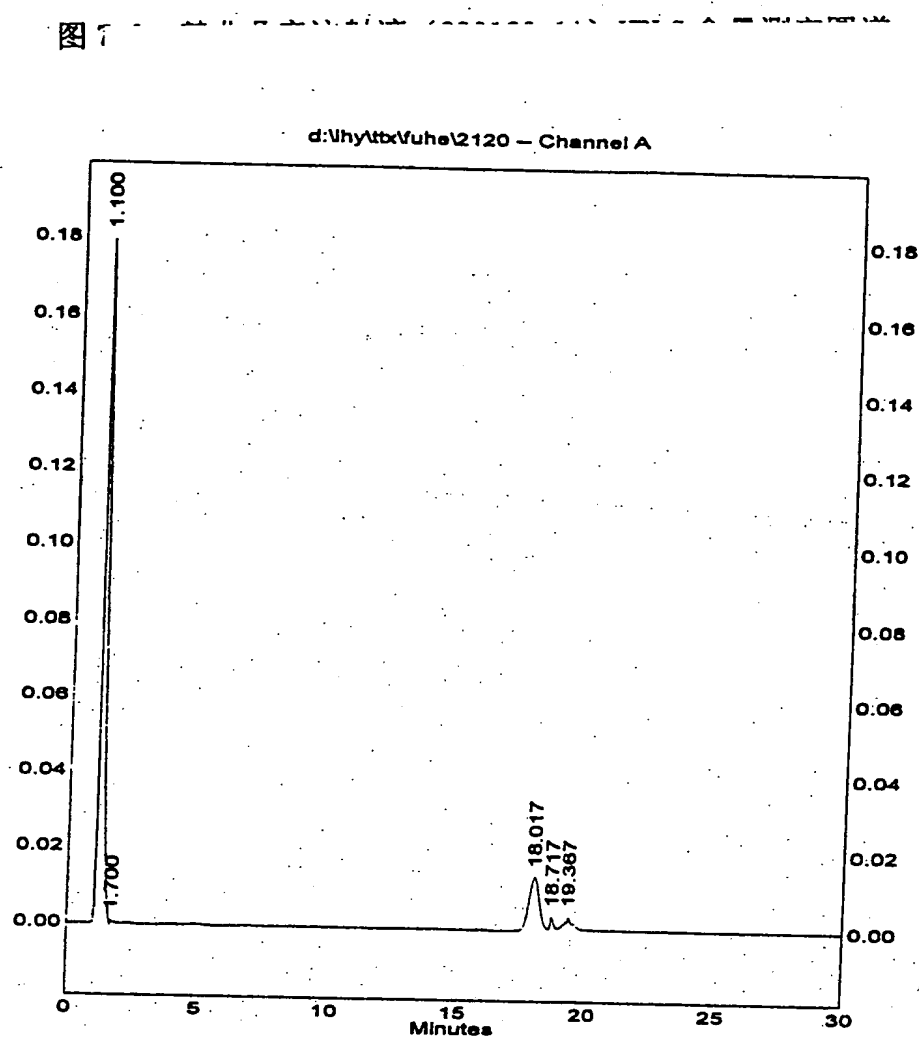
Control sample

Batch number 990120-1A

Batch number 990121-2A

Batch number 990122-3A

**Figure 6.** The HPLC chromatogram of content determination of tetrodotoxin injection (990120-1A).



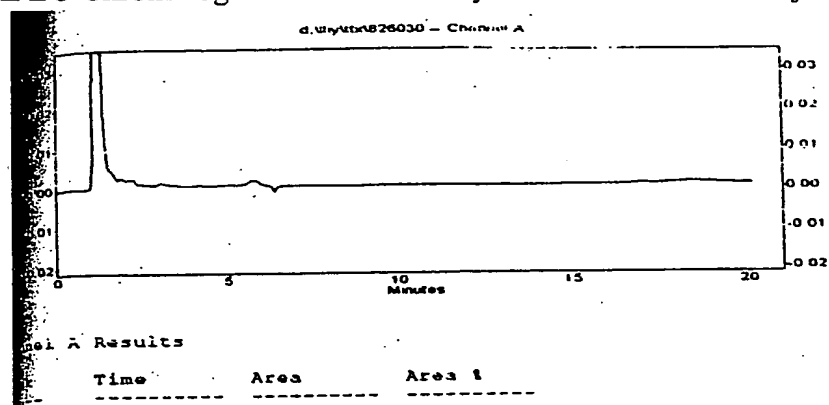
**Channel A Results**

Peak	Time	Area	Area %
1	1.10	2044956	80.199
2	1.70	10066	0.395
3	18.02	383153	15.026
4	18.72	30817	1.209
5	19.37	80869	3.172

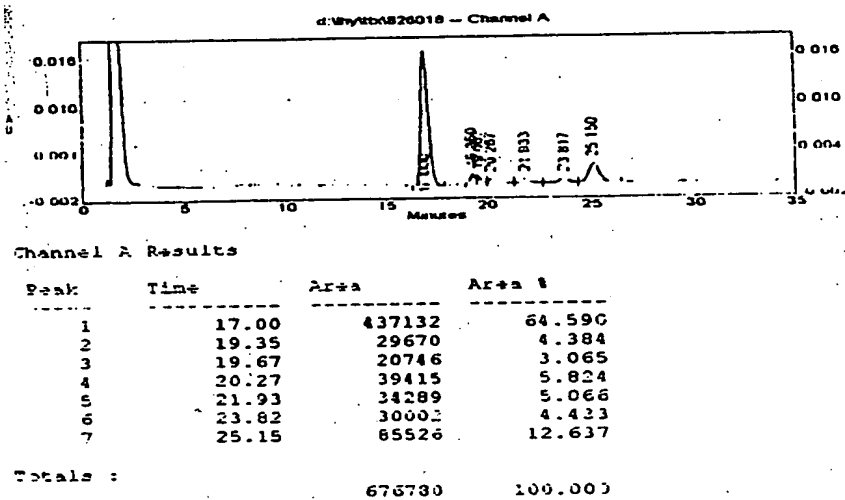
**Totals :**

2549861 100.000

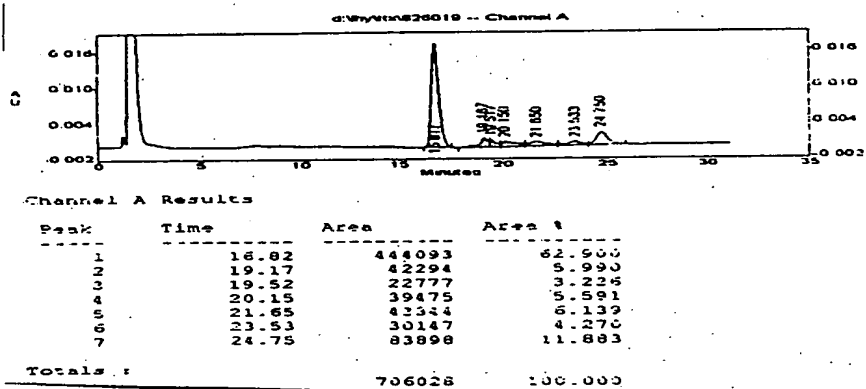
Figure 7. HPLC Chromatogram of the recovery test of Tetrodotoxin injection.



Solution A: Blank solution without tetrodotoxin prepared as the formula.

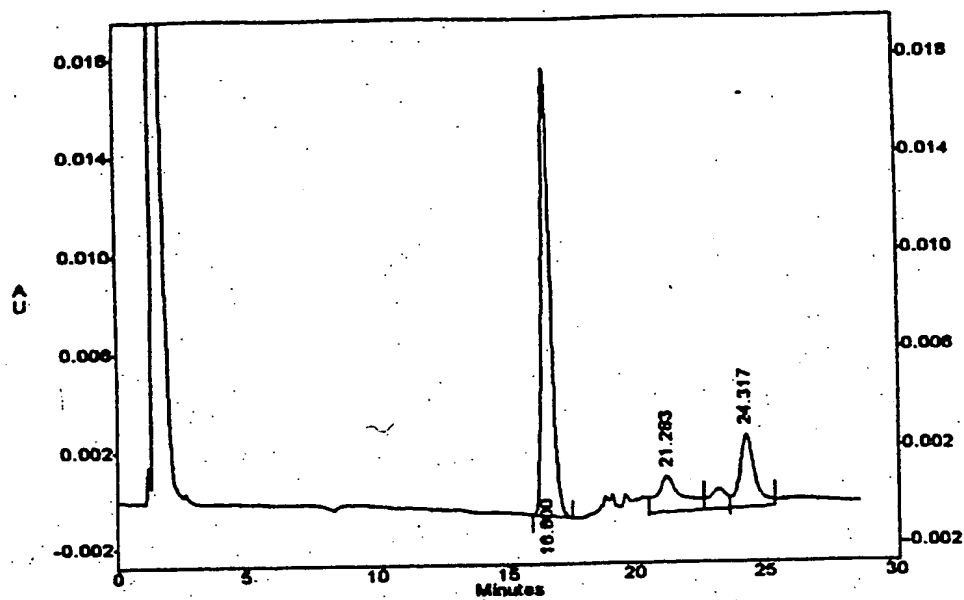


Solution C: The 0.2% acetic acid solution containing the primary pharmaceutical tetrodotoxin



Solution D: Solution A containing the primary pharmaceutical tetrodotoxin

Figure 8. Day 0 chromatogram of TTX injection samples (Batch 990120-1A).

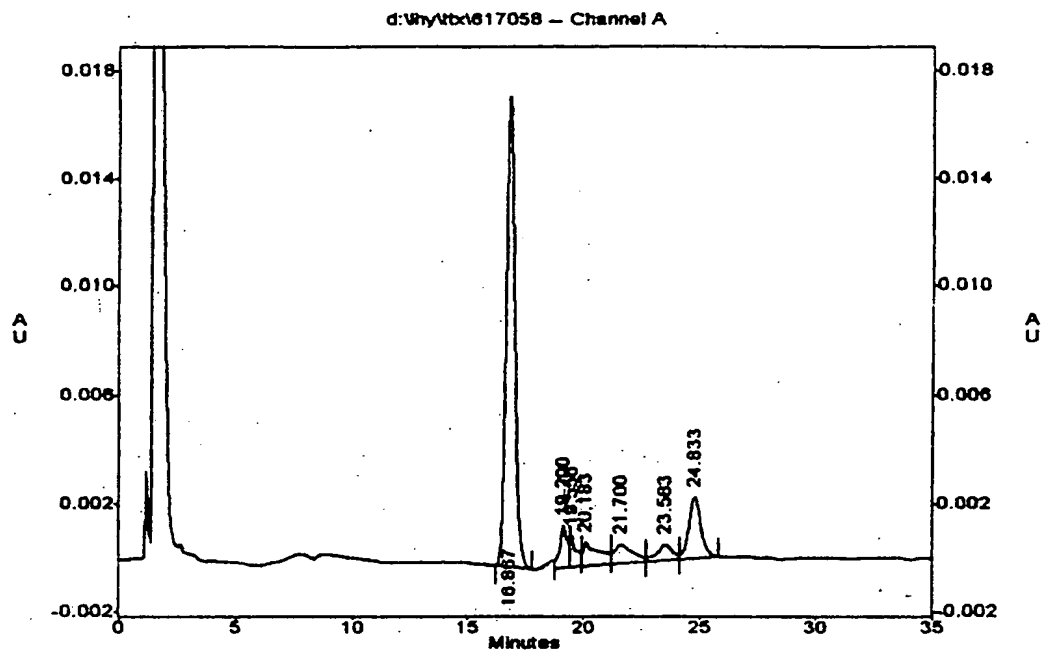


Channel A Results

Peak	Time	Area	Area %
1	16.60	457304	67.543
2	21.28	96642	14.274
3	24.32	123111	18.183
Totals :		677057	100.000



**Figure 9.** Day 5 chromatogram of TTX injection samples (Batch 990120-1A)  
exposed to fluorescence light



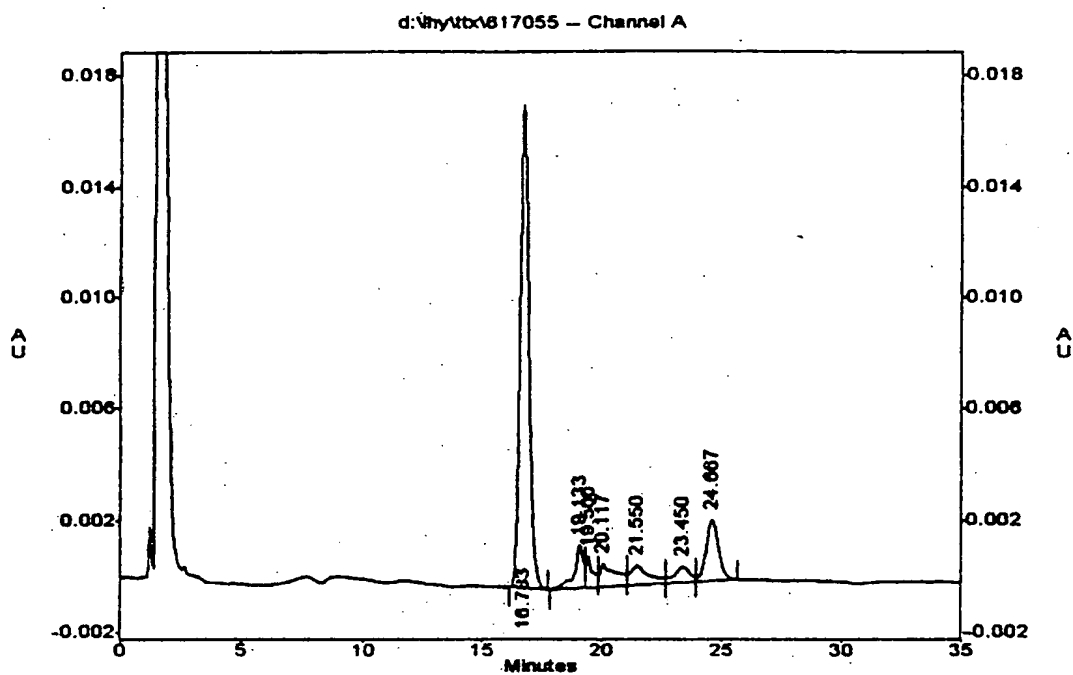
Channel A Results

Peak	Time	Area	Area %
1	16.87	439230	64.337
2	19.20	33194	4.862
3	19.55	21996	3.222
4	20.18	41813	6.125
5	21.70	38117	5.583
6	23.58	26835	3.931
7	24.83	81513	11.940

Totals :

682698 100.000

Figure 10. Day 10 chromatogram of TTX injection samples (Batch 990120-1A)  
exposed to fluorescence light



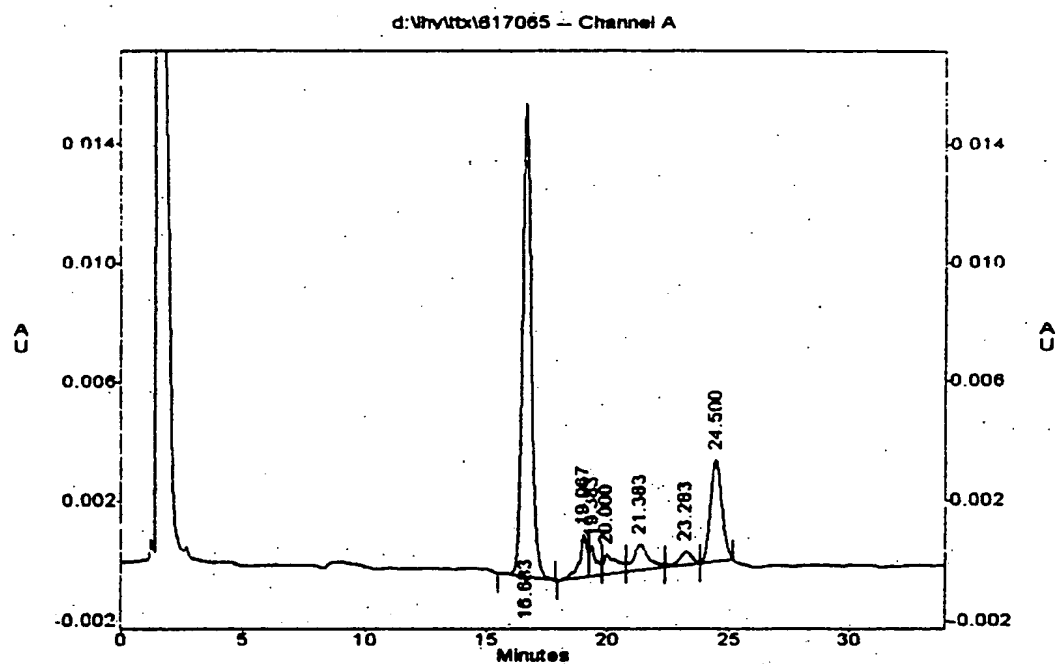
Channel A Results

Peak	Time	Area	Area %
1	16.78	440996	64.617
2	19.13	40110	5.877
3	19.50	21413	3.138
4	20.12	38546	5.648
5	21.55	38558	5.650
6	23.45	25316	3.709
7	24.67	77538	11.361

Totals :

682477 100.000

Figure 11. Day 5 chromatogram of TTX injection samples (Batch 990120-1A)  
stored at 40°C



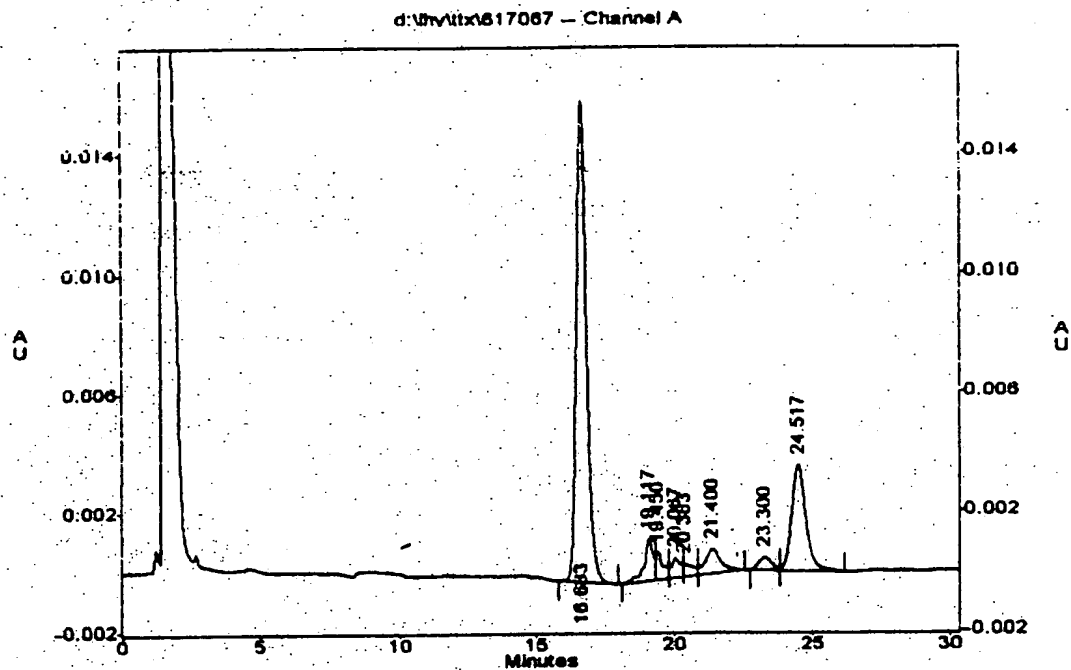
Channel A Results

Peak	Time	Area	Area %
1	16.68	403338	62.647
2	19.07	31695	4.923
3	19.38	18661	2.898
4	20.00	26281	4.082
5	21.38	37923	5.890
6	23.28	16412	2.549
7	24.50	109512	17.010

Totals :

643822 100.000

Figure 12. Day 10 chromatogram of TTX injection samples (Batch 990120-1A) stored at 40°C



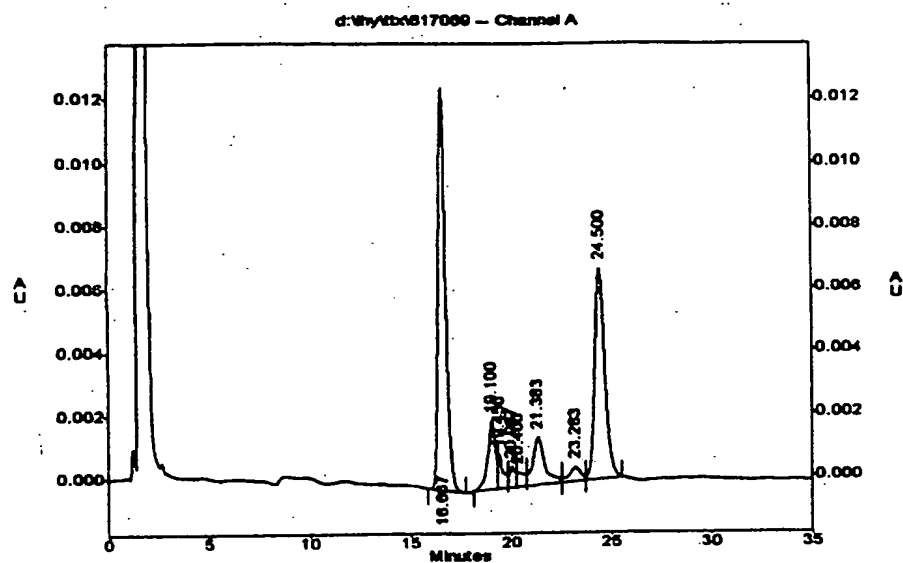
Channel A Results

Peak	Time	Area	Area %
1	16.68	406902	62.061
2	19.12	31196	4.757
3	19.45	16302	2.563
4	20.07	14841	2.264
5	20.38	10273	1.567
6	21.40	32718	4.990
7	23.30	14349	2.265
8	24.52	128073	19.534

Totals :

655644 100.000

Figure 13. Day 5 chromatogram of TTX injection samples (Batch 990120-1A) stored at 60°C



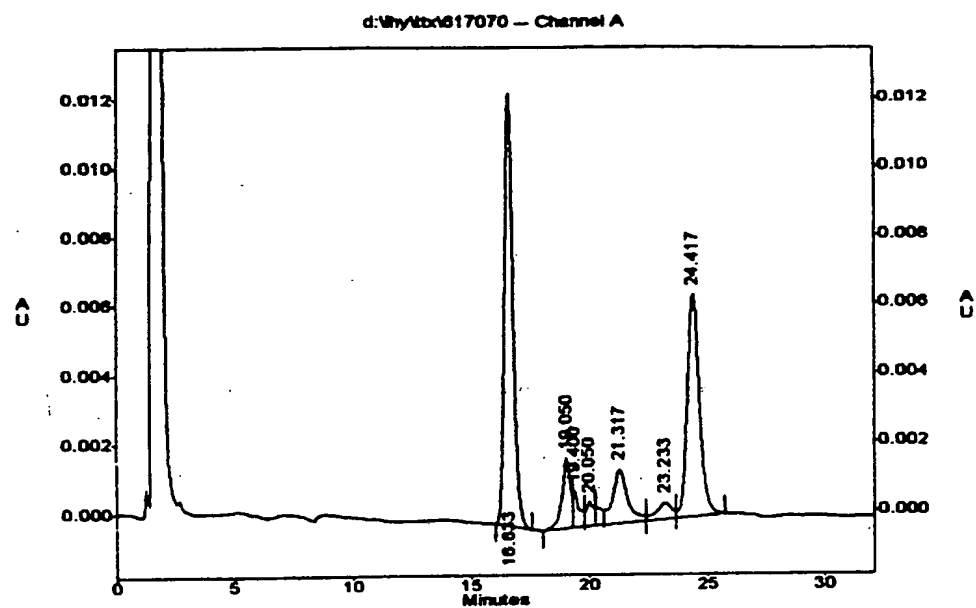
Channel A Results

Peak	Time	Area	Area %
1	16.67	320463	43.726
2	19.10	55618	7.589
3	19.45	20052	2.736
4	20.07	13250	1.808
5	20.40	13195	1.800
6	21.38	62082	8.471
7	23.28	17525	2.391
8	24.50	230698	31.478

Totals :

732883 100.000

Figure 14. Day 10 chromatogram of TTX injection samples (Batch 990120-1A)  
stored at 60°C

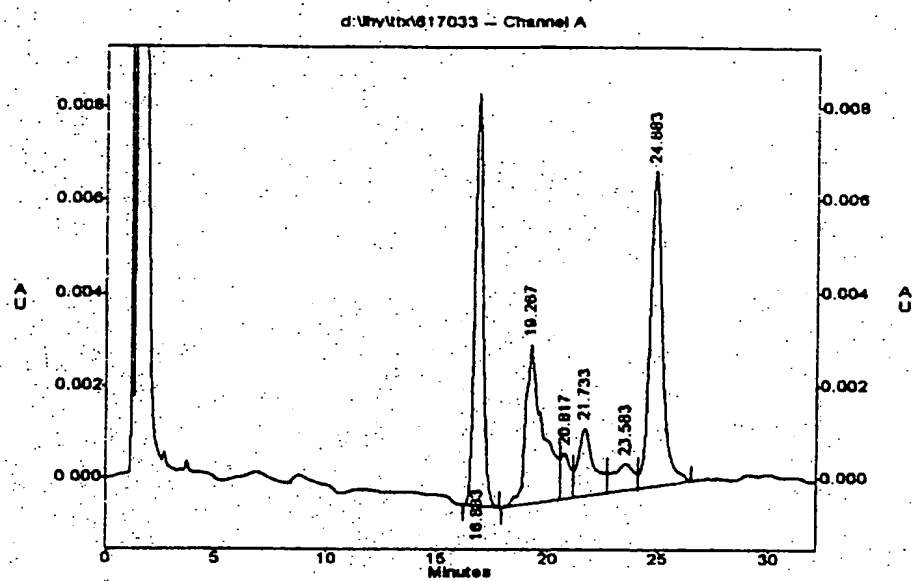


Channel A Results

Peak	Time	Area	Area %
1	16.63	312079	43.881
2	19.05	49723	6.992
3	19.40	20080	2.823
4	20.05	15408	2.167
5	21.32	67030	9.425
6	23.23	21349	3.002
7	24.42	225521	31.710

Totals : 711190 100.000

Figure 15. Day 5 chromatogram of TTX injection samples (Batch 990120-1A)  
stored at 80°C



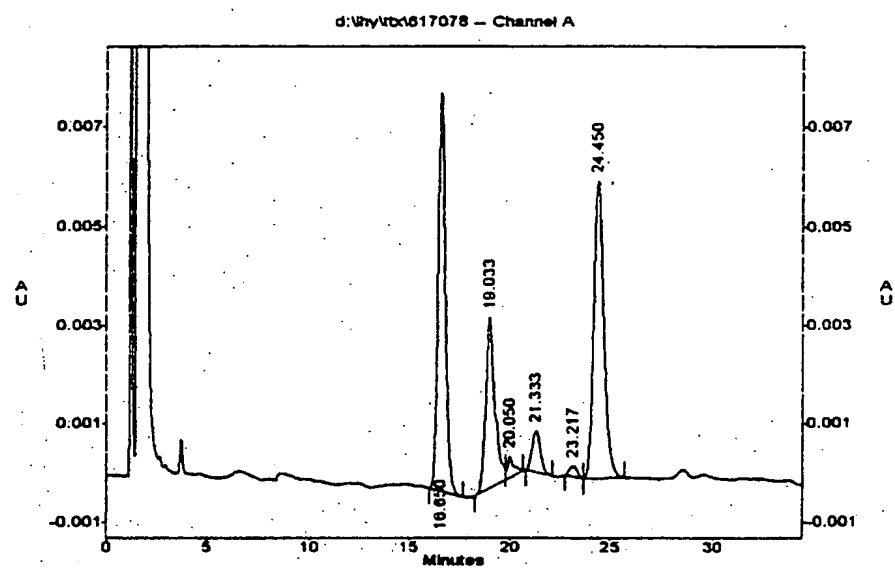
Channel A Results

Peak	Time	Area	Area %
1	16.88	225642	27.595
2	19.27	189762	23.207
3	20.82	29663	3.628
4	21.73	75156	9.191
5	23.58	37486	4.584
6	24.88	259987	31.795

Totals :

817696 100.000

**Figure 16.** Day 10 chromatogram of TTX injection samples (Batch 990120-1A) stored at 80°C



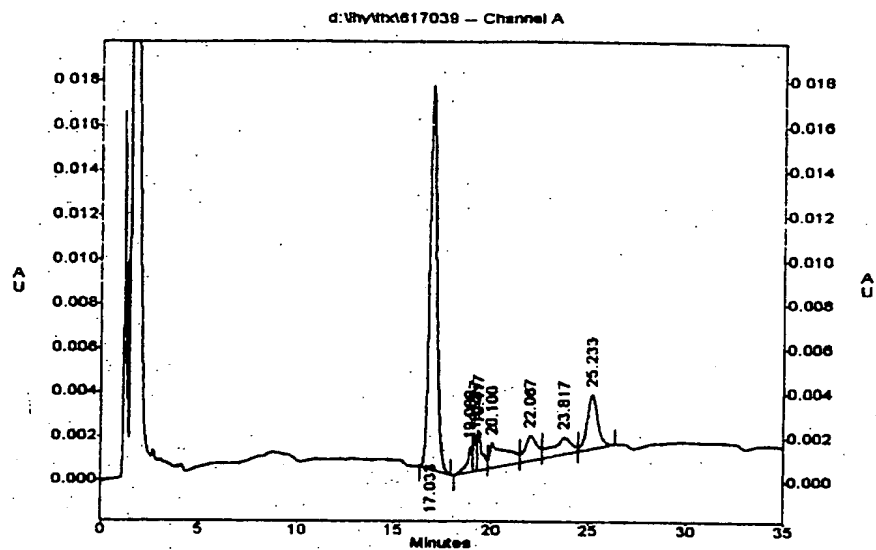
Channel A Results

Peak	Time	Area	Area %
1	16.65	201979	36.363
2	19.03	106755	19.220
3	20.05	9113	1.641
4	21.33	24854	4.475
5	23.22	5678	1.022
6	24.45	207069	37.280

Totals : 555448 100.000



Figure 17. Day 5 chromatogram of TTX injection samples (Batch 990120-1A) stored at the humidity of 75%

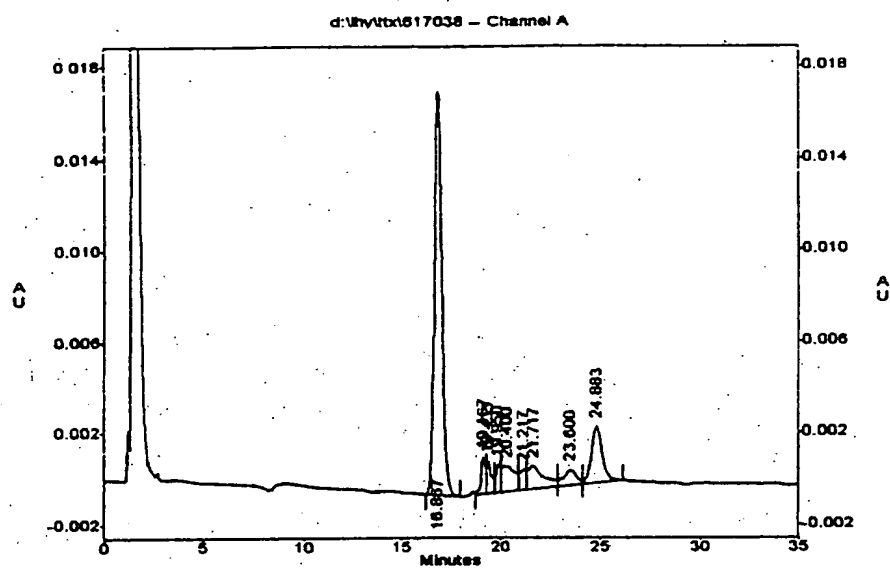


Channel A Results

Peak	Time	Area	Area %
1	17.03	440743	57.272
2	19.00	19869	2.582
3	19.22	16741	2.175
4	19.42	30163	3.920
5	20.10	65769	8.546
6	22.07	50126	6.514
7	23.82	52224	6.786
8	25.23	93927	12.205

Totals : 769562 100.000

Figure 18. Day 10 chromatogram of TTX injection samples (Batch 990120-1A) stored at the humidity of 75%

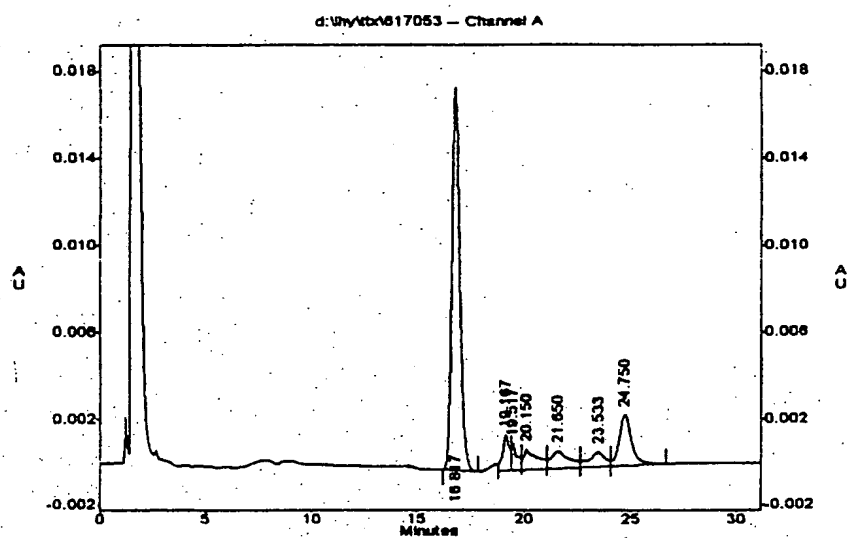


Channel A Results

Peak	Time	Area	Area %
1	16.87	447357	58.043
2	19.17	25793	3.347
3	19.43	23024	2.987
4	19.95	20972	2.721
5	20.40	50791	6.590
6	21.22	21996	2.854
7	21.72	56974	7.392
8	23.60	31567	4.096
9	24.88	92256	11.970

Totals : 770730 100.000

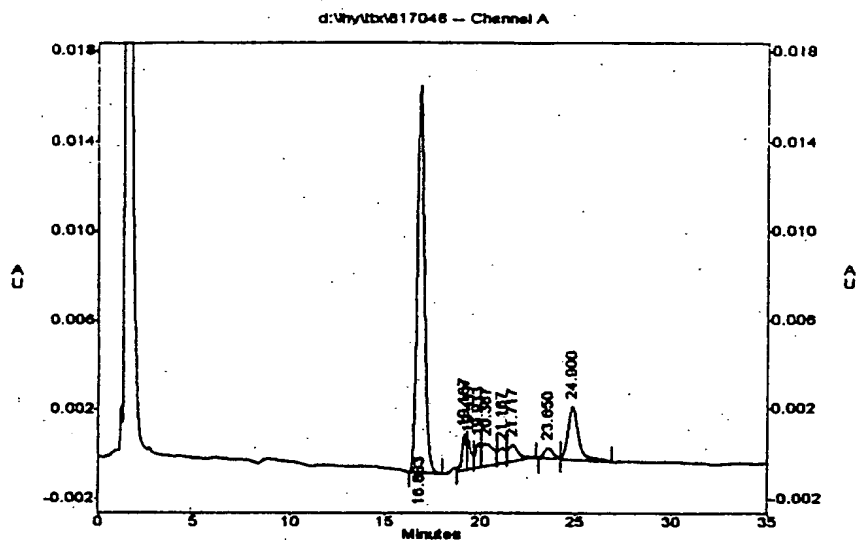
Figure 19. Day 5 chromatogram of TTX injection samples (Batch 990120-1A) stored at the humidity of 92.5%



Channel A Results

Peak	Time	Area	Area %
1	16.82	447441	61.629
2	19.17	34832	4.798
3	19.52	23418	3.226
4	20.15	41630	5.734
5	21.65	47836	6.589
6	23.53	35696	4.917
7	24.75	95165	13.108
Totals :		726018	100.000

**Figure 20.** Day 10 chromatogram of TTX injection samples (Batch 990120-1A)  
stored at the humidity of 92.5%

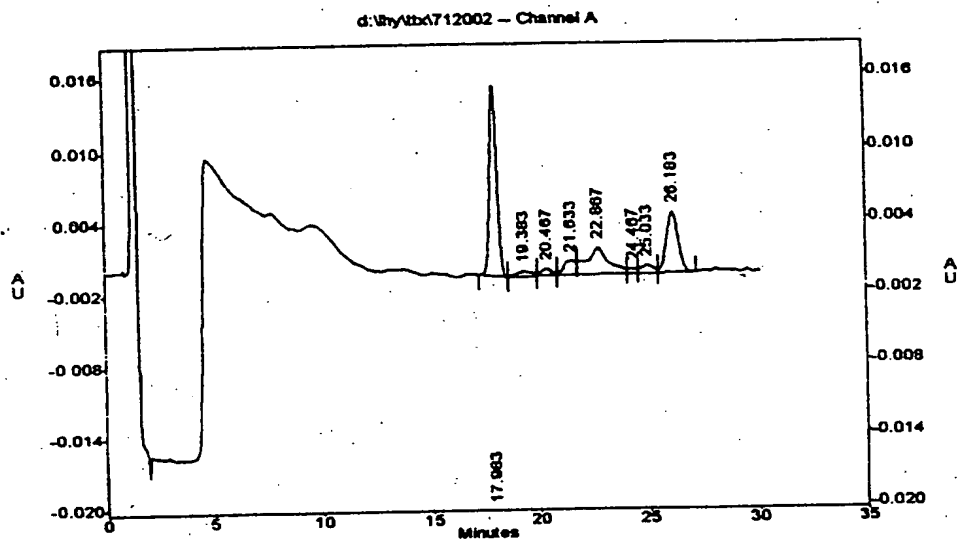


Channel A Results

Peak	Time	Area	Area %
1	16.88	439985	62.732
2	19.17	24726	3.525
3	19.42	20133	2.871
4	19.93	18443	2.630
5	20.37	43002	6.131
6	21.17	18768	2.676
7	21.72	29564	4.215
8	23.65	14957	2.133
9	24.90	91798	13.088

Totals :  
701376 100.000

Figure 21. Month 1 chromatogram of TTX injection samples (Batch 990120-1A)  
in acceleration test at 40°C



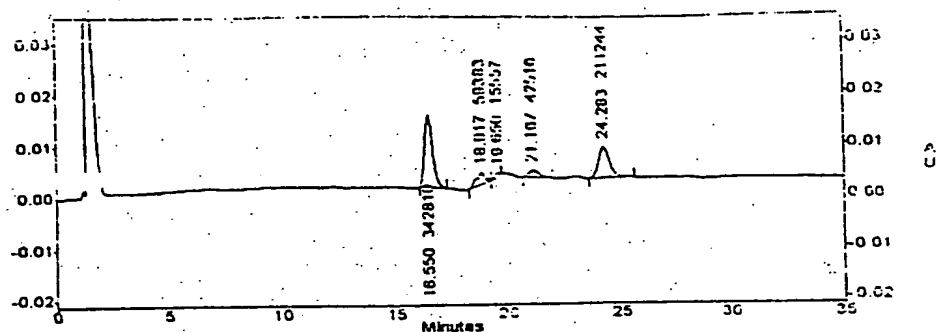
Channel A Results

Peak	Time	Area	Area %
1	17.98	376984	44.357
2	19.38	21365	2.514
3	20.47	22100	2.600
4	21.63	46498	5.471
5	22.87	157085	18.483
6	24.47	12261	1.443
7	25.03	28093	3.306
8	26.18	185497	21.826

Totals :

849883 100.000

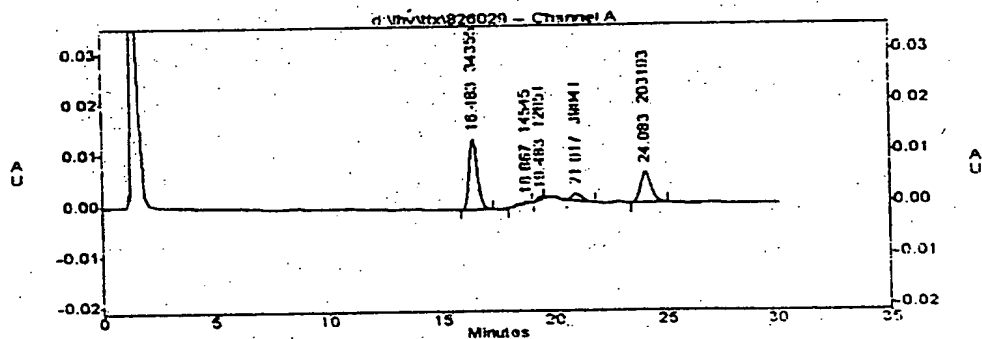
Figure 22. Month 2 chromatogram of TTX injection samples (Batch 990120-1A) in acceleration test at 40°C



Channel A Results

Peak	Time	Area	Area %
1	16.55	342810	51.051
2	18.92	59383	8.843
3	19.63	15557	2.317
4	21.17	42516	6.331
5	24.28	211244	31.458

Totals : 671510 100.000

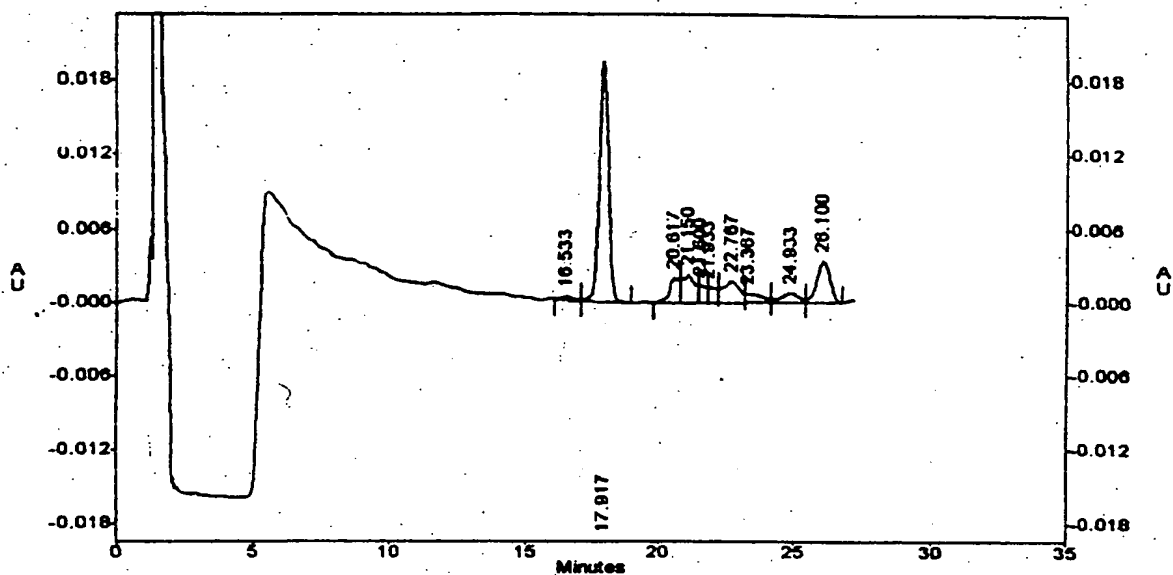


Channel A Results

Peak	Time	Area	Area %
1	16.48	343552	53.946
2	18.87	14545	2.269
3	19.43	12851	1.993
4	21.02	39941	6.504
5	24.08	203193	31.089

Totals : 614082 100.000

Figure 23. Month 1 chromatogram of TTX injection samples (Batch 990120-1A)  
stored at 4-5°C

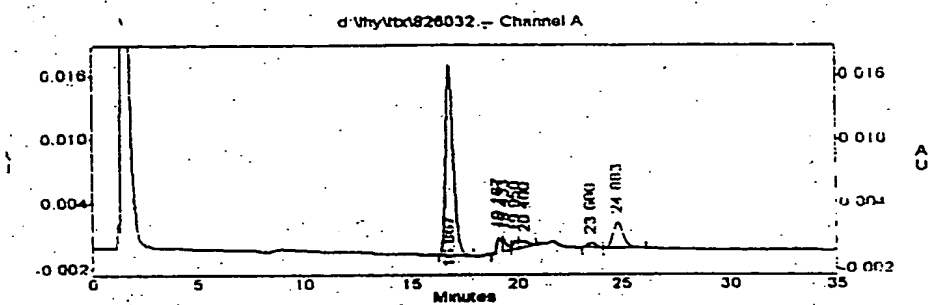


Channel A Results

Peak	Time	Area	Area %
1	16.53	12642	1.366
2	17.92	469198	50.691
3	20.62	53889	5.822
4	21.15	78215	8.450
5	21.60	28459	3.075
6	21.93	27149	2.933
7	22.77	75205	8.125
8	23.37	32292	3.489
9	24.93	38038	4.110
10	26.10	110521	11.940

Totals : 925608 100.000

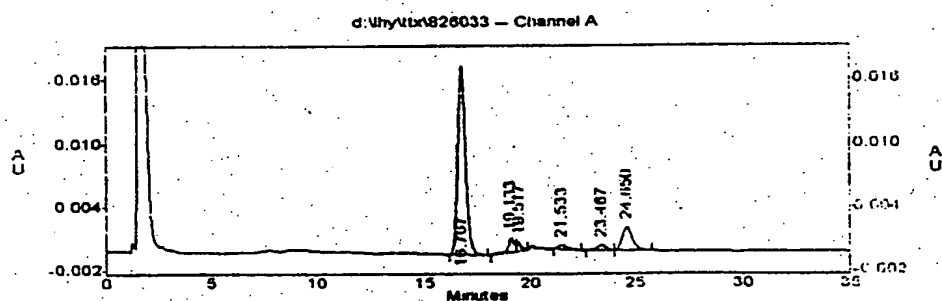
Figure 24 Month 3 chromatogram of TTX injection samples (Batch 990120-1A)  
stored at 4-5°C



Channel A Results

Peak	Time	Area	Area %
1	16.87	446937	73.398
2	19.17	19824	3.255
3	19.43	15306	2.513
4	19.95	11884	1.952
5	20.40	18910	3.106
6	23.60	13018	2.138
7	24.88	83086	13.644

Totals :  
608965 100.000



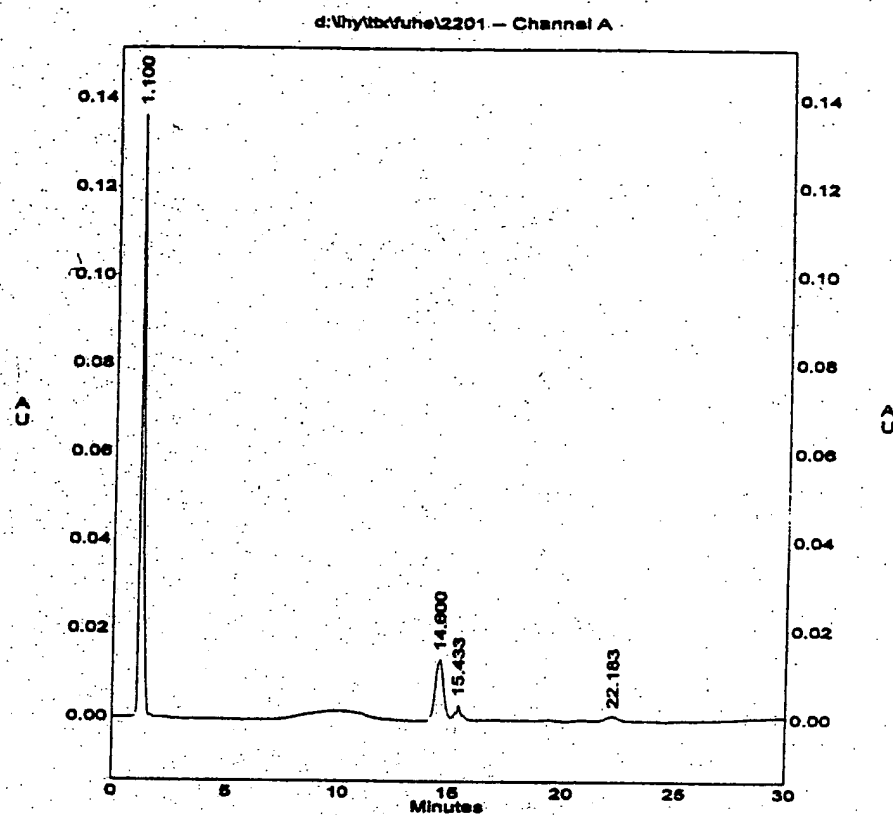
Channel A Results

Peak	Time	Area	Area %
1	16.77	447336	75.939
2	19.13	28063	4.764
3	19.92	10316	1.751
4	21.53	10525	1.787
5	23.47	15642	2.669
6	24.65	76939	13.070

Totals :  
589071 100.000



**Figure 25.** Month 6 chromatogram of TTX injection samples (Batch 990120-1A) stored at 4-5°C



Channel A Results

Peak	Time	Area	Area %
1	1.10	1329668	73.593
2	14.60	369604	20.456
3	15.43	67516	3.737
4	22.18	40001	2.214

Totals :

1806789 100.000